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The Development and Study of Organic Composites

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The Development and Study of Organic Composites

A Major Qualifying Project
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Abstract

To alleviate modern society's reliance on plastic, our team explored compostable alternatives to petroleum-based polymers. This study examines the processability and properties of organic fiber-reinforced composites, constructed using a combination of PLA or a potato starch-based matrix, and natural fibers such as flax, cotton, or jute. To characterize the resulting material, our team studied the matrix-fiber adhesion, chemical composition, mechanical strength, and biodegradability of the samples. This investigation allowed our team to infer potential applications for our product, as well as issue guidance for future exploration.

Executive Summary

Plastic is among the greatest contributors to the modern global waste crisis. To alleviate reliance on petroleum-based plastic materials, it is important to consider alternative options, such as bioplastics. Bioplastics are an increasingly popular alternative to petroleum-based plastics for their more responsible sourcing and ability to decompose. However, bioplastics alone are not a solution to all applications, particularly not ones that require specific mechanical properties such as high strength. One way to fulfill this need is with composites, where another material is added to the bioplastic to manipulate the properties of the resulting material.

This study examines the processability, properties, and degradability of an organic fiber-reinforced plastic composite. Specifically, we created composites with natural fibers such as flax, cotton, and jute as well as biodegradable resins such as PLA and potato starch-based plastics. To understand these composites, they were tested in their capacity to degrade in different environments, as well as examined on a microscopic level. Results and recommendations for further exploration into these topics will be provided.

The goal of this project was to create a material that had a lower environmental impact than petroleum-based plastics without sacrificing desirable mechanical properties. We had two objectives to meet our project goal:

- 1.) To develop a novel composite material made from bioplastic resins, namely potato starch-based plastics or PLA, reinforced with natural fibers such as flax, jute, and cotton
- 2.) To explore the properties of the composite including matrix-fiber adhesion, degradability, material composition, and mechanical strength

Our team first looked into fibers and resins which may be suitable for this type of project. Based on availability, pricing, and background research to make sure options were biodegradable, it was concluded that PLA and potato starch-based plastics would be explored with raw cotton, flax, and jute fibers. Molds were developed for the composite to be formed in, and several environments were considered for this process including an oven, a hot press, a vacuum pressure vessel, and open air. It was determined that the best option would be to synthesize potato starch-based plastic, impregnate it with random chopped fibers, and allow it to cure in the aforementioned environments. Once composites were obtained, they were examined using SEM analysis. Then, some selected samples were placed into ocean water, a compost and dirt mixture, and a low-oxygen environment so that their decomposition could be monitored for weeks. FTIR analysis and Instron mechanical property testing were planned, but could not be completed due to the outbreak of the COVID-19 pandemic. Mechanical properties and FTIR results for the composites were estimated instead. Lastly, the fiber density of the composites was analyzed to determine trends for the relationship between density and decomposition rates.

Composite samples with a potato starch-based resin and flax or cotton fibers show very good matrix-fiber adhesion and minimal flaking. Samples with a potato starched based resin and jute fibers, however, did not show the same level of matrix-fiber adhesion. It is hypothesized that the samples with less flaking and better matrix-fiber adhesion would have higher tensile strengths.

Although FTIR analysis could not be conducted, the spectra for potato powder, raw potato, potato puree, potato puree with additives, and flax fibers were examined. We believe that the results of FTIR analysis on our samples would share similarities with these spectra.

We planned to conduct Instron testing to determine the mechanical properties. Our molds yielded rectangular results but would have been laser-cut to the specified dogbone shape necessary for Instron tests. However, since this was not able to be conducted, existing mechanical property information for the components of our composites was used to make the following predictions:

- 1.) The Young's Modulus (YM) of our composite samples can be roughly characterized as the mean of the YM of its components.
- 2.) The ultimate tensile strength is roughly that of the matrix component.
- 3.) The yield strength approximately reflects that of the matrix component.
- 4.) The composite will not be as ductile as the matrix component; it will likely reflect the brittle nature of the fiber component, despite similar fracture loading as the matrix.

Materials that most closely resemble the strength-to-density ratios of our samples included carbon foam, vermiculite, aluminum 5052 honeycomb, concrete, and bricks. These materials have similar void fractions to the ones seen in our composites when observed in our SEM analysis.

PLA was explored, but ultimately not used to create composite samples. Baking the plastic from pellet form in a mold resulted in a sample with many bubbles and a viscosity too high to allow for fiber impregnation. Adding solvents to the pellets while melting did not decrease the viscosity or produce a homogeneous sample. The polymerization of PLA with fibers in-situ was also explored but was not able to be performed. The best method was determined to be pulverizing the PLA into a powder, impregnating the powder with random chopped fibers, and melting the sample in a hot press.

The density of samples was determined via a simple mass-volume relationship of the overall sample. The sample with the lowest density was a high flax composite, with a density of 0.787 g/mol. The low density and high fiber content are likely related, as the fibers used have much lower densities than the resin. Determining the density was used to reveal a relationship between density and degradation rates.

In a compost environment, the high flax composite showed the fastest rate of degradation. There is indeed a correlation between density and degradation as evidenced by the

data collected; density and degradation rates are inversely proportional. In ocean water, the high flax sample broke down well; it became swollen opaque while losing mass, which is indicative of the breaking of polymer bonds. This suggests true compostability. Finally, samples also had comparable degradation rates in low-oxygen environments, similar to those that may be found buried in a landfill.

Based on our findings, several recommendations were developed to provide future researchers with a variety of avenues to explore on this topic:

We recommend characterizing the mechanical properties of potato starch-based fiber-reinforced composites.

To better understand the composites and create an informed profile of them, Instron testing is necessary. We recommend that samples are shaped into the specific dogbone specimen shape required for the Instron test either by a dog bone-shaped mold or laser-cutting a rectangular sample.

We recommend identifying the composition of potato starch-based fiber-reinforced composites.

FTIR analysis is recommended to obtain a better profile of the composition of the composites. This is important for exploring potential applications and determining the relationship between the makeup of the composites and the composites' other properties.

We recommend curing fiber-reinforced composites under vacuum conditions to minimize voids within the sample.

Higher void rates within samples appear to correspond to faster rates of degradation. It is important to learn to manipulate this factor to consider the materials for more applications.

We recommend creating samples with different fiber orientations, resins, and fiber types.

Specifically, we believe it would be beneficial to explore corn starch-based resins, as well as other biodegradable fibers in orientations such as unidirectional or woven.

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1.0 Introduction

Among the greatest global engineering challenges faced today is the waste crisis which is exacerbated by modern society's reliance on synthetic plastics. Synthetic petroleum-based plastics are especially common in the automotive and aeronautical industries, but at the end of their useful life, these materials are difficult to recycle and contribute greatly to the waste crisis. Plastics synthesized using petrochemicals include polyethylene, PVC, polypropylene, polystyrene, polyester, and nylon, and these materials require hundreds to thousands of years to fully decompose in a landfill [1]. Regarding petroleum-based plastics and their impact on the environment, author and researcher David Barnes suggests that "one of the most ubiquitous and long-lasting recent changes to the surface of our planet is the accumulation and fragmentation of plastics," [2]. Furthermore, these highly durable, yet destructive plastics are in limited long-term supply, and therefore, more extensive research into organic, biologically-sourced plastics is critical to building a sustainable future.

Biologically-sourced plastics, or bioplastics for short, are, "one of the fastest-growing sectors of the plastics industry, with an anticipated 20-30% annual growth," [3]. By definition, these materials are synthesized from renewable resources while maintaining their recyclable and biodegradable characteristics [4]. Although some petroleum-based materials are biodegradable, their degradation rates are significantly lower when compared directly to bioplastics. Additionally, bioplastics could soon be very cost-efficient alternatives to their petroleum-based counterparts. According to the European Polymer Journal, the "expected continuation of high crude oil and natural gas prices... allow bioplastics to become more cost-competitive with petroleum-based resins," [4].

To emphasize the need for an expansive bioplastics industry, it is important that the waste crisis on Earth is simultaneously examined. In *The Voice of Clean Capitalism*, Adria Vasil asserts that cargo ships filled with plastics are actively being turned away by global authorities in Asia, and roughly nine percent of all plastic produced is recycled [5]. Given these staggeringly low recycling rates and limited global accountability for plastic waste, a large majority of solid plastic waste remains neglected in landfills. Decomposition of plastic waste is typically very slow, but as these landfills accumulate more waste, oxygen exposure becomes very limited, and degradation is hampered significantly. The BBC news reports that the United States, "...produces three times the global average of waste, including plastic..." [6]. However, bioplastics could positively impact the waste crisis significantly. Environmentalist Paul Hawken estimates that replacing up to 49% of all plastics with bioplastics, "could save a whopping 4.3 gigatonnes of carbon (equivalent to taking 1 billion cars off the road for one full year) by 2050." [5].

Although bioplastics, such as PLA and potato plastic (proposed in this study), are a necessary part of slowing future environmental catastrophe, they may not be suitable for all applications by themselves, primarily ones requiring advantageous mechanical properties. Thus,

natural fiber-reinforced composites could play a key role in solving this problem. In these applications, bioplastics serve as a resin material which is then combined with one or multiple natural fibers to produce an organic biocomposite.

The goal of our project is to develop novel composite materials made from biodegradable resins, reinforced with natural fibers. Beyond the development of fiber-reinforced composites, we will examine how each sample is processed, as well as provide detailed procedures for replicable results. Matrix-fiber adhesion, fiber orientation, chemical composition, mechanical strength, and biodegradability of our composite material will all be investigated. This investigation will not only allow our team to infer potential applications for our product but also hopefully encourage change toward a more sustainable, carbon-neutral future.

As a result of the rapid spread of the COVID-19 virus, experimentation and data collection for this project ceased on March 6, 2020 by WPI due to government mandates. This paper includes information and experimentation leading up to that date, as well as a description of activities that we had hoped to conduct after that date.

2.0 Background

2.1 Material Categorization

‘Environmentally friendly’ materials typically fall into one or more of the following categories: biodegradable, compostable, or organic. The general public’s ability to distinguish between these categories is often unreliable due to their overuse as buzzwords in greenwashing campaigns [7]. However, scientific communities have stringent classification requirements when it comes to the categorization of green materials.

Biodegradable materials are those which break down into basic elemental components in the presence of microorganisms, such as those found in natural environments. These materials decompose into smaller units and lose properties associated with the original material. This is not to be confused with degradable materials, which also break down into elemental components, but through a variety of different means often involving light and heat [8].

A compostable material is similar to a biodegradable material in terms of the process in which it breaks down. However, a material is considered to be compostable only if it can achieve total degradation into environmentally safe components. These materials do not leave behind toxic traces, even in elemental form [8]. The process of composting produces carbon dioxide, water, inorganic compounds, and biomass. It is important to note that compostable materials are biodegradable, but not all materials that are biodegradable are compostable.

Organic materials, by definition, are compounds which are carbon based. These compounds were initially observed as being found in the natural environment, however, with modern technology Carbon based compounds can also be synthesized in a laboratory setting. These organic compounds include petroleum based plastics, and other synthetic materials which are also composed of Carbon [9]. Natural organic materials are those which are chemically unaltered from their naturally sourced form. Organic materials include plant fibers and natural starches. Organic matter is compostable unless it has been combined with other materials that prevent complete degradation into nontoxic units.

In this paper, “biodegradable” refers to a material that breaks down into smaller components in the environment. These smaller components may contain Persistent Organic Pollutants (POPs) or other toxins, and they may have the potential to bioaccumulate. “Compostable” materials are those which break down into smaller components in the environment and do not pollute the environment or cause health issues for local flora and fauna. “Organic” materials are those which are unaltered and sourced from the environment, and therefore are likely compostable. The intention of this project was to develop a fiber-reinforced composite with desirable mechanical properties which is both organic and compostable.

2.2 Bioplastics

Bioplastics, including polylactic acid (PLA) and polyhydroxyalkanoates (PHA), are polymers which are generated from the polymerization of natural monomers. These monomers can be harvested from sources such as plants, microorganisms, and animals [8]. However, many bioplastics are created using naturally sourced starch monomers in combination with artificially synthesized polymers. Despite the potential inclusion of synthetic polymers, bioplastics are biodegradable. However, the inclusion of synthetic materials, such as petroleum-based chemicals, often prevents the polymer from biodegrading into entirely environmentally safe components.

2.3 Degradation

The factors which most affect a material's rate of degradation include nutrient composition, pH, gas exchange capabilities and the temperature of the environment [10]. By measuring biodegradation under optimal conditions, the intrinsic biodegradability of the material is determined. When placed in less than optimal conditions, even the most biodegradable material may not show promise.

Optimal soil nutrient composition, pH, and gas exchange rates are used to achieve optimal conditions for target microorganisms. Different microorganisms are better suited to combat different pollutants. For this reason, remediation techniques such as bioaugmentation, bioremediation, and biostimulation alter soil conditions to create environments which encourage the growth of microorganisms which incite the biodegradation of a particular pollutant. More specifically, bioaugmentation is the process of introducing microorganisms to a polluted area, and may be used to initiate degradation of materials which were not breaking down in the environment in which they were introduced [8]. Bioremediation is similar to bioaugmentation, however, this typically involves the more strategic selection of targeted microorganisms in addition to microbial nutrients. Biostimulation, on the other hand, is dependent upon an existing microbial population which is stimulated through the addition of optimal nutrients.

Despite all other factors which influence biodegradation, it seems that biodegradation is impacted the greatest by temperature, and that the relationship between the temperature and the rate follows the Arrhenius equation [11]. Therefore, to favor the growth of mesophilic microorganisms, degradation studies should be conducted at moderate temperatures between 20C and 28C.

$$k = Ae^{\frac{E_A}{RT}} \quad (\text{Eq. 2.2.1})$$

Where: k is the rate constant

A is the pre-exponential factor

e is the mathematical quantity

R is the gas constant

T is the absolute temperature in Kelvin

EA is the reaction activation energy

There are two industry standards for measuring the degree and rate of aerobic biodegradability of plastics in soil - the International Standard ISO 17556 and ASTM D 5988. These standards involve studying measured quantities of carbon dioxide over time to determine the degree of aerobic biodegradation [12]. However, environmental fate studies are possible to execute by measuring the mass loss of polymers in soil burial experiments.

Of greatest concern are the conditions found in landfills, bodies of water, and the natural environment.

2.4 Composites

Composite materials are those made from two or more separate materials with varying properties to produce a single new material with its own unique properties. There are a variety of composite classifications including laminar composites, particulate composites, and fiber composites [11]. Concrete is the most common example of a particulate composite; it is made from a cement matrix reinforced by aggregate, or loose stone, particles [11]. Carbon fiber and fiberglass are common examples of fiber-reinforced composites and may be found in applications such as bicycle frames or automotive bumpers, respectively. The increasing popularity of composites can be attributed to their high strength-to-weight ratios, durability, and cost-effectiveness as production methods evolve [13].

Of particular interest to us are fiber-reinforced composite materials consisting of two primary structures: a fiber and a matrix. First, the fiber or filament material primarily enhances the strength and stiffness of the product [14]. Additionally, the reinforcing fiber can contribute to the corrosive resistance and rigidity of the composite. All of these properties, however, are directly related to the orientation of the fiber [15]. Likewise, the matrix structure functions to “bind the fiber reinforcement, transfer loads between fibers, give the composite component its net shape and determine its surface quality.” [16]. Composite matrices typically display favorable shear properties and low density [17]. Matrices can exist as ceramics, metals, or even polymers, and of particular interest to us are potato plastic and polylactic acid resin matrices. Operating in tandem, the fiber and matrix structure is critical to the performance of the product.

For example, the adhesion between the fiber and resin matrix is the primary mode of stress transfer in the composite, resulting in favorable mechanical properties [18].

2.5 Effect of Fiber Quantity on Mechanical Properties

Regarding mechanical properties, fiber-reinforced biocomposites, “have been introduced into the market because of the increasing demand for advantageous mechanical properties per weight” [4]. Fiber-reinforced composites, first, demonstrate a high tensile strength. According to one research journal, the tensile strength of fiber-reinforced composites increases proportionally with the fiber content of the material [19]. Similarly, the Young’s Modulus increases with heightened fiber loading [19]. However, there is “a maximum or optimum value,” of fiber that can be inserted into the composite until the mechanical properties start to decrease, and the theoretical tensile strength and Young Modulus can be predicted using the Rule of Mixtures and Halpin–Tsai equations respectively. [19]. Other properties expressed by fiber-reinforced composites include high stiffness and toughness [18].

2.6 Effect of Fiber Orientation on Mechanical Properties

The mechanical properties of fiber-reinforced composites are not only related to the quantity of fibers in the composite, but also the orientation of fibers within the composite. A study on fiber orientation conducted in 2010 states that there are three fundamental types of fiber structures: random, unidirectional, and woven fabric [18]. Furthermore, this study concluded that “unidirectional fiber possesses higher tensile strength and module compared with woven and random mat fibers, but woven fiber offers higher flexural strength and module, whereas random mat shows poor properties for all loading conditions compared with woven and unidirectional fibers” [18]. This behavior is very similar to that of chopped and continuous fibers being implemented into 3D printed parts. In chopped fiber composites, the fibers are reduced to small fragments and combined with the plastic in order to improve properties such as, “strength, stiffness, and dimensional stability of the part to make it higher-performing than its base plastic” [20]. In continuous fiber composites, strands of fibers are input into the plastic matrix without interruption, maximizing the materials load capacity and impact absorptivity to magnitudes rivaling high strength metals [22].

Composites with random fibers do not have the same levels of high tensile strength, but have the advantages of being the easiest to produce [15]. Since the need for maintaining parallel fibers in the matrix is eliminated, fibers may simply be chopped and combined with the polymer. Their random nature creates isotropic properties, as forces applied from different directions should have fibers supporting the matrix in each direction. This method requires significantly less effort than woven fibers, as well. The result is a material with strength, although not as high

as in other orientations, in every direction that is easy to manufacture quickly and on a large scale [15].

2.7 Mechanical Testing Strategies

Although the mechanical properties of fiber-reinforced composites can be generalized in comparison to traditional materials, there are several important tests that must be conducted in order to determine the properties of a specific material. The core mechanical properties of a material that must be tested in a laboratory, and are often done on a tensile testing machine, include ultimate tensile strength, modulus of elasticity, yield strength, Poisson's ratio, and strain rates. All are determined via tensile testing, specifically the ISO 527-4 test [21], on an Instron machine. This involves acquiring a dimensionally specific piece of the fiber-reinforced composite and clamping it into a machine that pulls each end of the specimen until the specimen fails (see **Figure 2.7.1**).

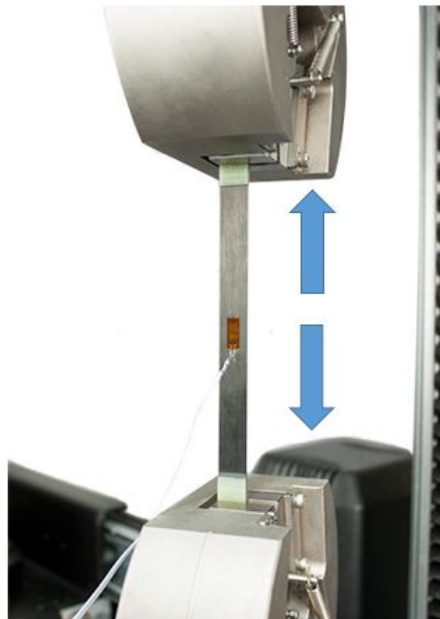


Figure 2.7.1. The specimen, seen in the center of the image, is pulled according to the direction represented by the blue arrows [21].

A strain gauge is attached to the outside of the specimen in order to measure the rate of elongation at given stress rates. The machine is programmed to constantly measure and record changing values of strain, stress, and applied forces in order to calculate the mechanical properties of the specimen. For a fiber-reinforced composite, the proper specimen shape is classified as a Type 1B, also called a “dogbone,” which has a rectangular cross-section, and whose overall shape can be seen in **Figure 2.7.2** [21].

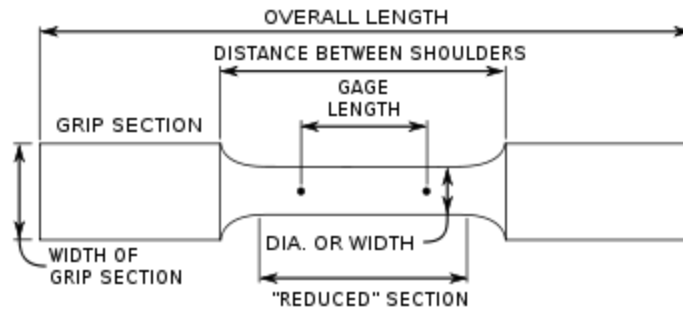


Figure 2.7.2. The overall shape of a “dogbone” tensile test specimen.

2.8 Current Applications of FRPs

The attractive mechanical properties and cost-effectiveness of fiber-reinforced plastics, or FRPs, has led to a massive increase in their usage worldwide. FRPs offer high strength-to-weight ratios, high stiffness-to-weight ratios, low fatigability rates, high impact tolerances, and high compressive and tensile strengths [22]. Almost a third of FRP use is accounted for by the automotive industry [23]. This is largely because of the strength-to-weight ratios of FRP, which are of the utmost concern in automotive applications in order to optimize speed and fuel efficiency. The second largest consumer of FRPs, for similar reasons, is the construction industry, where they are used to retrofit and increase the load-bearing capacity of existing structures such as columns or beams, repairing damage to existing structures, or producing things like bridge decks and pipe fittings [23]. Some FRPs have resistance to corrosion, and thus make great candidates for applications involving marine environments. However, a naturally-sourced, biodegradable composite would be best for applications where corrosion is not a factor, and the product is eventually wasted, such as sports equipment, musical instruments, furniture, and household appliances.

2.9 Polylactic Acid (PLA)

Polylactic acid, or PLA, is a common type of bioplastic. It is sourced from natural materials such as cassava, sugarcane, or most commonly, corn [24]. PLA can be used in applications such as lost PLA casting, packaging in manufacturing, degradable medical implants in the biomedical field, prototyping via 3D printing, and many others. The advantages of PLA include not only its renewable sources and many potential applications, but its potential for degradability. In conditions with high heat and microorganisms to feed on the natural building blocks of PLA, it can begin to show signs of degradation within 6 months [24]. Most

conventional plastics such as polyethylene, polypropylene, and polystyrene are not biodegradable at all. These factors make PLA a good candidate for a thermoplastic matrix in a fiber-reinforced biocomposite.

2.10 Starch-based Plastic

Starch-based plastics are one of the most promising new areas for the development of sustainable, biodegradable plastics. Starch is completely biodegradable, inexpensive, and renewable. Some of the most common sources of starch include corn, potato, sweet potato, tapioca, and wheat [25]. For our project, we chose to focus on potato starch. Potato starch consists of two different polymers, amylose and amylopectin. Amylose is a straight chain of glucose units which can be used to form plastic. Amylopectin is a branched glucose polymer that inhibits the formation of plastic. The amylopectin must be broken down using hydrochloric acid to make smaller, straight-chain glucose polymers [26].

Starch alone can be used to make plastic, but the result has poor physical properties. They are brittle when dry and have poor mechanical properties when wet. They are also hydrophilic and have a tendency to recrystallize. One way to improve the mechanical properties while retaining the plastic's biodegradability is to add a natural plasticizer such as glycerol, glycol, or sorbitol. Another method is to add starch to natural or synthetic polyesters, like PLA, to create a starch blend that is completely biodegradable. Starch has also been added to nonbiodegradable, commodity plastics like polyethylene or polystyrene to improve biodegradability [25].

The market for starch-based bioplastics is rapidly growing. There are already several companies that produce these bioplastics, and many potato processing factories are changing their production line to reclaim potato starch released when the potatoes are cut [27]. Due to their poor mechanical properties, they are often used for packaging like cups, bowls, and bottles [25]. Some companies, like BiologiQ, are even using their plastic to make bags to hold their potatoes [27]. The main setback for potato plastic is the cost. Production costs are slightly higher or about even with the cost of fossil fuel-based plastics with a lower quality product [27]. In order for potato plastic to compete with petroleum plastics in the market, their production price will have to be lower.

3.0 Methods

To develop novel samples of compostable, organic composites, we:

- 1.) Gathered information regarding the availability of fiber and resin materials
- 2.) Developed a mold design for standardized sample specifications
- 3.) Synthesized potato-starch and PLA-based plastics using different fibers as reinforcement
- 4.) Extensively tested the macroscopic and microscopic characteristics of our material
- 5.) Conducted a degradation study of on our final composite samples

3.1 Fiber and Resin Selection

Before producing our composite material, our team needed to gather information regarding fibers and resins that could be integrated into our composite. Information that was of particular interest to us was the component type, the unit amount being offered, and the price per unit. By synthesizing information gathered from websites such as Walmart, Alibaba, Michaels, and Amazon, **Tables 3.1.1** and **3.1.2** were created to display information for fibers and resins respectively:

Component	Unit Amount	Unit Price
Flax	4 ounces	\$10.00
Banana Fiber	1, 2, or 4 ounces	\$(3.15-9.35)
Jute Fiber	10 lbs	\$24.00
Sisal	125 grams	\$5.00
Sisal Twine	150 feet (length)	\$2.16
Cotton	1 kilogram	\$1.17

Table 3.1.1. Fiber availability research table.

Component	Unit Amount	Cost
PLA	5 kg	\$47.17
TPS	25 kg	\$(3.70-4.00)
PHA (polyhydroxyalkanoates)	1 kg	\$(3.20-4.00)
PBS (polybutylene succinate)	1 kg	\$(1.00-99.00)
PCL (polycaprolactone)	50 g	\$1.89
Potato plastic	22 ounces	\$4.39

Table 3.1.2. Resin availability research table.

From this, our team deduced that the fibers most reasonable in size and cost were raw cotton, flax, and jute. PLA and potato-starch were the most suitable resins for our project going forward. One factor not listed in our tables that played a significant role in our decision-making was availability. Lots of these materials, especially resin materials, are only available in bulk overseas shipments while others are not commercially available at all. We initiated several phone calls with plastic fabrication companies in the Worcester, MA area to discuss the availability of these resins, and as result, were able to successfully locate our resin materials at a reasonable price.

3.2 Mold Development

Next, we developed a mold that could be used to create uniform samples of our final material. The method of sample fabrication is known as casting. Several design considerations went into the creation of our final product. We decided that our mold would be designed to completely enclose our sample during the curing process, thus a separated base and cover were required.

The base of the mold must primarily contain a rectangular bore for the composite to rest during the drying or curing process. The base of the mold was seven inches in length and four inches wide with a 4 x 2 x 0.5-inch borehole directly in the center. This borehole must include acutely angled walls, known as drafting angles, for ease of part removal. The cover of the mold contains four threaded holes (one in each corner) and a 4 x 2 x 0.1-inch boss that is toleranced to fit within the base's borehole. Overall, the mold was 1.5 inches tall without including the additional height accounted for by the hex bolts. A drawing sheet of our final mold assembly created in Solidworks can be seen below in **Figure 3.2.1**:

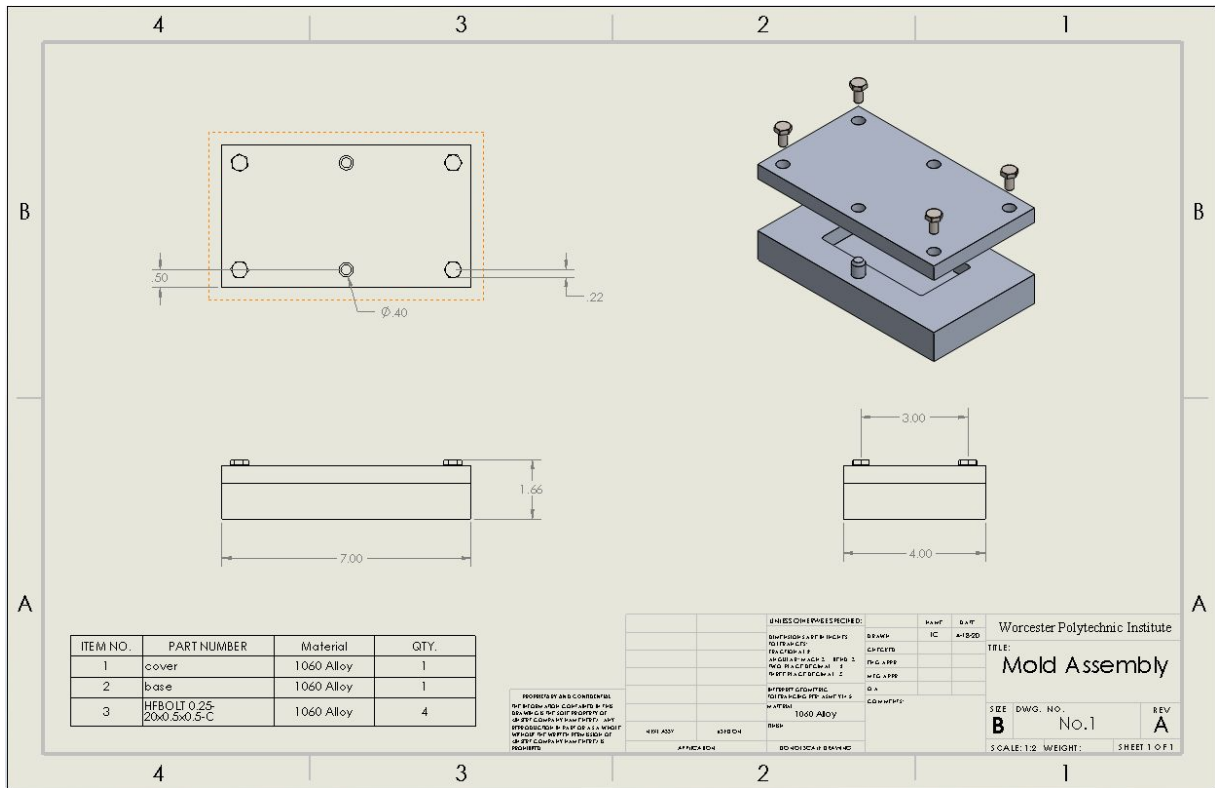


Figure 3.2.1. Drawing sheet for rectangular mold configuration.

The material used to create this mold was 1060 aluminum alloy. A Bill of Materials can be found in the lower-left corner of this drawing sheet. Tom Partington, the machine shop and lab director in WPI's chemical engineering department assisted in the design and CNC machining of our parts. Images of our base, cover, and assembly from the Solidworks design window as well as the mass properties of each can be found in **Appendix A**.

3.3 Synthesis of a Starch-Based Matrix

The first step in creating organic FRCs was to synthesize the starch-based matrix. Once this was complete, the matrix could either be cured as an organic polymer sample or combined with organic fibers to create a composite. The materials and equipment required for this process can be seen in **Table 3.3.1** below.

Materials	Equipment
<i>Potato Starch</i> <i>Deionized Water</i> <i>Propan-1,2,3-triol (Glycerol, Lab Grade)</i> <i>0.1M Hydrochloric Acid</i> <i>0.1M Sodium Hydroxide</i>	<i>Hot Plate</i> <i>100 mL Beaker</i> <i>Stirring Rod</i> <i>Mass Balance</i> <i>Weigh Boat</i> <i>Oven</i>

Table 3.3.1. Materials and equipment table - synthesizing starch-based matrix.

The procedure shown for synthesizing our potato starch-based matrix is as follows:

- 0) Prepare solutions of *0.1M hydrochloric acid* and *0.1M sodium hydroxide*, if only standard solutions are available.
- 1) Fill a *100 mL beaker* with 25 mL of *deionized water*.
- 2) Measure 2.5 g of *potato starch* with a *mass balance* and add it to the *deionized water-filled beaker*.
- 3) Add 3 mL of *0.1M hydrochloric acid* to the solution.
- 4) Add 2 mL of *propan-1,2,3-triol (glycerol, lab grade)* to the same on.
- 5) Place the solution on a *hot plate* at 200 C for typically 15-30 minutes.
- 6) Stir regularly with the *stirring rod*.
- 7) Remove the *beaker* from the *hot plate* once the mixture becomes viscous and the potato starch has been completely dissolved, and add 3 mL of *0.1M sodium hydroxide* to the solution to neutralize it. Stir briefly.
- 8) Allow the solution to cure overnight in an oven overnight at 105 C or leave uncured for fiber-reinforced composite preparation.
- 9) Clean all materials used in the lab.

3.4 Preparation of Fiber-Reinforced Composites

To create fiber-reinforced samples, the synthesized starch-based matrix was combined with varying quantities and types of organic fibers. These samples were then cured in multiple different environments, to test the variations in composite properties when produced under different processing conditions. To begin this process, the following materials, seen in **Table 3.4.1**, were prepared:

Materials	Equipment
<i>Starch-Based Matrix (uncured) Organic Fibers</i>	<i>Mass Balance Stirring Rod Beaker Aluminum Mold 70 mm Disposable Aluminum Tray Scissors Oven (if testing various process conditions) Vacuum Pressure Vessel (if testing various process conditions)</i>

Table 3.4.1. Materials and equipment table - preparation of FRCs.

The following procedure was used to develop the fiber-reinforced composite samples:

- 1) Cut each *organic fiber* to lengths of approximately 0.5 - 1.0 cm.
- 2) Using a *mass balance*, measure *organic fiber* to the amount of 0.25 g, 0.5 g, and 0.75g.
- 3) Place the *organic fiber* into a *70 mm disposable aluminum tray* or *aluminum mold* depending on the prospective testing method. Samples obtained from the *70 mm disposable aluminum tray* can be tested using the Scanning Electron Microscope (SEM) and Fourier-Transform Infrared Spectroscopy (FTIR) analyses. Samples created in the *aluminum mold* can be used for Instron testing.
- 4) Pour the *uncured starch-based matrix* into the tray or the mold and stir well.
- 5) Repeat steps 3 and 4 for all fibers and fiber densities.
- 6) Place each sample in an oven overnight at 105 C, or place in a vacuum pressure vessel at room temperature for three days.

3.5 Scanning Electron Microscopy (SEM) Analysis*

The first material analysis performed was a Scanning Electron Microscope (SEM) analysis. The SEM was used to observe the adhesion between the fiber and the plastic matrix as well as look for any other noticeable features in the composite. **Table 3.5.1** shows the materials used for this process.

Materials	Equipment
<i>Composite Sample</i> <i>Gold</i> <i>Aluminum Stub</i> <i>Sticky Carbon Disk</i>	<i>Sputter Coater</i> <i>Scanning Electron Microscope (SEM)</i>

Table 3.5.1. Materials and equipment table - SEM analysis.

The following procedure was used to obtain SEM pictures of our composite samples:

- 1) Make sure the *composite sample* is completely clean and dry before using the *SEM*. The sample can be put back into the oven at 105 C for a couple hours to ensure it is dry.
- 2) Sputter coat the *composite sample* in *gold* to increase the conductivity of the sample.
- 3) Place the sample on the *aluminum stub* using a *sticky carbon disk*.
- 4) Vent the vacuum chamber of the SEM before opening it. Once it has been vented, place the sample into the vacuum chamber and close the door.
- 5) Turn on the vacuum and start the electron beam. Using microscope software on the SEM's computer, the focus, magnification, and location on the sample can be adjusted. If the image is still not focused, the sample height can be adjusted as well.
- 6) Once the images of the sample have been taken at several different magnifications and locations, turn off the electron beam, vent the vacuum chamber, and remove the sample.
- 7) Repeat steps 1-6 for all samples [28].

3.6 Fourier - Transform Infrared Spectroscopy (FTIR) Analysis**

Fourier-Transform Infrared Spectroscopy (FTIR) is an analytical technique used to identify the composition of a material. It obtains an infrared spectrum of absorption or emission which can be used to verify the expected material as well as identify any contaminants. The materials needed for FTIR analysis are shown in **Table 3.6.1**.

Materials	Equipment
<i>Acetone</i> <i>Matrix Sample</i> <i>Kimwipe</i>	<i>FTIR</i>

Table 3.6.1. Materials and equipment table - FTIR analysis.

The procedure for obtaining compositional data is as follows:

- 1) Turn on the *FTIR* and the computer. Open the FTIR software on the computer.
- 2) Wipe the sample holder with *acetone* and a *Kimwipe* to remove any contaminants.
- 3) On the computer, click the “background” button to collect the background spectra without the sample.
- 4) Place the *matrix sample* on the sample holder. Tighten the clamp until the force gage is around 80. Press the “Start” button to begin the scan.
- 5) Once the scan has finished, loosen the clamp, remove the sample, and clean the sample holder with acetone and a Kimwipe. Shut down the computer and the FTIR [29].

3.7 Instron Testing to Determine Mechanical Properties**

To determine the mechanical properties of a sample of fiber-reinforced composite, an Instron testing machine was required. This machine, as detailed in the background section, tests for fundamental mechanical properties by applying tensile forces to the sample, typically until it breaks. The sample must be prepared before the test, however, to dimensions specified by Instron standards. To do this, a mold with the specified shape of the test specimen was created, or as in the case of this research, a rectangular mold was used to create a sample of fiber-reinforced composite larger than the required specimen size. These samples were to be cut in a laser-cutting machine, available in the MakerSpace on campus at WPI, to fit the dimension requirements for the Instron test.

Materials	Equipment
<i>Prepared fiber-reinforced composite sample</i>	<i>Instron tensile testing machine</i>

Table 3.7.1. Material and equipment table - Instron testing.

The procedure for Instron testing machines depends on the machine model itself and the guidelines of the department in charge of the machine, but are as follows:

- 1) Turn on the Instron machine and the computer. Open the Instron software on the computer.
- 2) Place the sample into the bottom grip of the Instron machine and clamp it in place.
- 3) Lower the top grip of the Instron machine toward the specimen and clamp the specimen into place.
- 4) Check to make sure the specimen is well clamped in the center of the grips and is as close to vertical as possible.
- 5) Apply the strain gauge to the “gauge length” portion of the test specimen.
- 6) Use the computer software to conduct the Instron tensile test and record data.

3.8 Density Determination

Our team sought to determine the density of our different samples, so we could see how these properties relate to the rates of degradation observed.

Materials	Equipment
<i>Composite Sample(s)</i> <i>DI Water</i>	<i>Mass Balance</i> <i>Graduated Cylinder, 200 mL</i> <i>Thermometer</i> <i>Calculator</i>

Table 3.8.1. Material and equipment table - density determination.

The procedure for determining the density of our samples was as follows

- 1.) Determine the mass of all composite samples when dry. Record.
- 2.) Determine the error associated with the mass balance and graduated cylinder combination
 - a.) Determine the mass of the larger graduated cylinder, by weighing it when empty. Tare the scale, and leave the graduated cylinder on the balance.
 - b.) Measure out 150 mL DI water into the graduated cylinder, and record the mass.
 - c.) Determine the temperature of the water using the thermometer, and locate the density of water at that temperature. Calculate the expected mass of 150 mL of water using the density for that temperature.
 - d.) Compare the actual mass vs the theoretical mass to determine the combined error of the measuring devices.

$$\% \text{ Error} = \frac{|\text{Accepted Value} - \text{Experimental Value}|}{\text{Accepted Value}} * 100$$

- 3.) Submerge a single composite sample into the graduated cylinder. Record the volume change, and ensure that the new mass is as expected.
- 4.) Remove the composite sample, and set aside.
- 5.) Calculate the density of the sample, assuming uniform distribution throughout the sample
$$\rho = \frac{\Delta \text{mass}}{\Delta \text{Volume}}$$
- 6.) Repeat steps 2-5 for each sample being tested.

3.9 Degradation of an Organic Composite

Our team sought to determine the rate at which our organic composites degrade in a variety of different conditions. We focused on open-air natural environments, high salinity bodies of water, and anaerobic environments. The materials and equipment required for this process can be found in **Tables 3.8.1 and 3.8.2.**

Materials	Equipment
<i>Composite Sample(s)</i> <i>Organic Compost</i> <i>Red Wigglers</i> <i>Organic Matter</i> <i>Ocean Water</i> <i>Saccharomyces Cerevisiae</i> <i>Water</i>	<i>Vapor Lock</i> <i>3 Large Vessels (approx. 4 liters)</i> <i>Plumber's Putty</i>

Table 3.9.1. Materials and equipment table - degradation study.

Our team developed three different degradation chambers which were utilized to study how our composites broke down in a variety of different environments. These different environments included: compost, ocean water, and a low oxygen organic environment.

The procedure for creating a compost degradation chamber was as follows:

- 1.) Clean and dry a large vessel (glass or plastic) with a volume of approximately 4 liters
- 2.) Fill the vessel with approximately 3 liters of soil
- 3.) Add newspaper, low acid organic matter (celery tops, potato peels, egg shells, etc), and earthworms (Red Wigglers)
- 4.) Add composite sample, water once per week, and keep in an environment with a temperature of approximately 68°F.

The procedure for creating an ocean water environment was as follows:

- 1.) Clean and dry a large vessel (glass or plastic) with a volume of approximately 4 liters
- 2.) Fill the vessel with approximately 3 liters of ocean water
- 3.) Add composite sample, and keep in an environment with a temperature of approximately 68°F.

The procedure for creating a low oxygen organic environment was as follows:

- 1.) Clean and dry a large vessel (glass or plastic) with a volume of approximately 4 liters
- 2.) Drill a hole in the cap of the vessel, just large enough to push a vapor lock through. Seal the area around the vapor lock using Plumber's Putty to prevent airflow.
- 3.) Fill the vessel with approximately 3 liters of soil.
- 4.) Activate yeast according to Manufacturer's directions, and ensure yeast is active before introduction to environment.
- 5.) Add composite sample, cap, fill vapor lock

- 6.) Water once per week and keep in an environment with a temperature of approximately 68°F.

Materials	Equipment
<i>Composite Sample(s)</i> <i>Degradation Chamber(s)</i>	<i>Mass Balance</i> <i>Oven</i> <i>Paint Brush</i> <i>Forceps</i>

Table 3.9.2. Materials and equipment table - mass measurement.

All samples were cleaned and allowed to dry before the mass of each was recorded. The procedure for the sample preparation prior to data collection was as follows:

- 1.) Using forceps or chopsticks, remove sample from degradation chamber. Ensure a complete sample is retrieved. If the sample is in multiple pieces, collect all pieces.
- 2.) Rinse any large debris off the composite sample.
- 3.) Dry samples in a single layer in the oven at 70°F for 24 hours.
Alternatively***: Dry samples in a single layer in the oven at approximately 100°F for 75 minutes, or until the sample is dry to the touch. (Note: the thin flexible composite samples will take 50% less time, and ocean water samples will take approximately 50% more time.)
- 4.) Using paint brushes, brush any remaining dirt and debris off samples to ensure an accurate reading. If moisture is present under debris, continue drying until the sample is dry to the touch.
- 5.) Weigh sample, and record. Return sample to degradation chamber.

*This process was completed by Doug White, who provided our team with the resulting SEM images.

** Results were not acquired from this test, due to a combination of technical issues, and the unexpected COVID-19 lockdown of campus prior to the end of the research term.

*** Alternate drying method deployed once access to the lab was no longer available. The reason for this alternate method is because there was no oven available which could achieve temperatures as low as 70°F.

4.0 Results

4.1 *Potato Plastic Fiber-Reinforced Composite Synthesis*

The primary focus of our study was potato starch fiber-reinforced composites. In *Section 3.4*, we describe the process of synthesizing our composite samples. The result was three composite samples with a uniform 0.25 g fiber density seen below in **Table 4.1.1**.




Fiber Density	Flax-Reinforced	Jute-Reinforced	Cotton-Reinforced
0.25 g			

Table 4.1.1. Composite Sample Images.

We next created samples with increased fiber densities, specifically 0.5 g and 0.75 g for each fiber type. Due to the COVID-19 pandemic, we were unable to obtain images for these samples. Once we completed our experimentation, we looked beyond physical characteristics to gain a better understanding of these foundational composite samples.

4.2 *SEM Analysis*

Pictures of our composite samples were taken at several different magnifications using an SEM. One of the main goals was to observe the adhesion between each type of fiber and the potato plastic matrix. Images were taken for composites made with jute, flax, and cotton. Imaging the PLA with flax sample was attempted, but the surface was too smooth for the SEM to take a picture of.

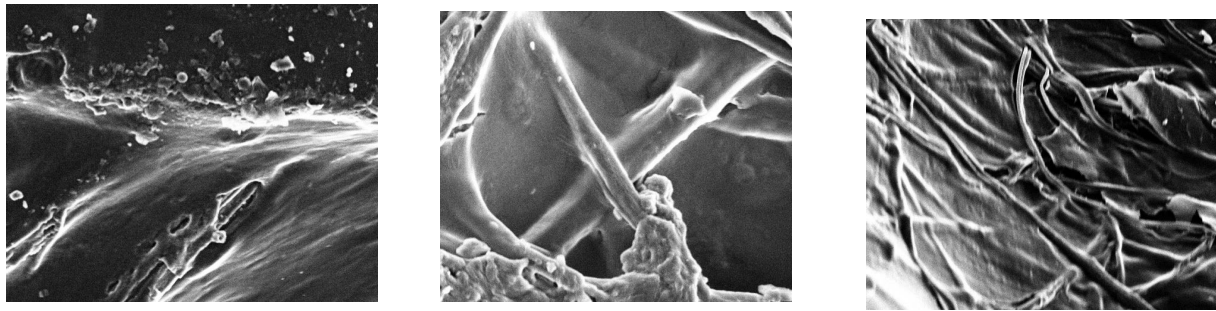


Figure 4.2.1. Jute, 250X (left); Flax, 500X (center); Cotton, 250X (right)

Figure 4.2.1 shows pictures of the three different types of fiber used. Noticeably, the flax and cotton samples have very good fiber-matrix adhesion. There are no flakes of the matrix in the pictures. The jute sample, however, shows worse fiber-matrix adhesion. At the top of the image, there is flaking of the matrix which shows it did not adhere as well to the fiber. While we were unable to perform strength testing on our samples, it is hypothesized that samples with better fiber-matrix adhesion, like the flax and cotton samples, would have higher tensile strengths than the jute sample.

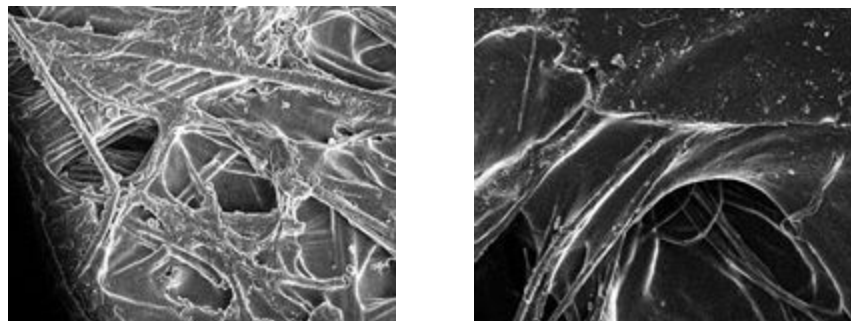


Figure 4.2.2. Flax, 250X (left); Jute, 50X (right)

One of the other noticeable features of some composite samples is presence of pores between the matrix and fiber. **Figure 4.2.2** shows flax on the left and jute on the right, both of which have visible pores. The cotton sample did not have these pores, as seen in **Figure 4.2.1**. While holes in a composite may seem like deformity, they can be useful for stopping the propagation of cracks. In addition, we suspected that these pores may be useful for the degradation of the material. These pores allow spaces for microorganisms to thrive which can increase the rate of degradation. An increased rate of degradation may not be desirable for some applications of the composite, so the composite was cured in the presence of a vacuum to remove the pores. Due to time limitations, we were unable to obtain SEM images of the vacuum sample.

The sample seemed to be much denser than our other samples, so it is hypothesized that it has fewer voids and would degrade at a slower rate.

4.3 FTIR

An FTIR analysis was planned for our composite samples but was unable to be performed due to time restrictions. However, the results of other studies can be used to predict the spectra of our samples. **Figure 4.3.1** shows the FTIR spectra for potato starch, potato puree, and raw potato.

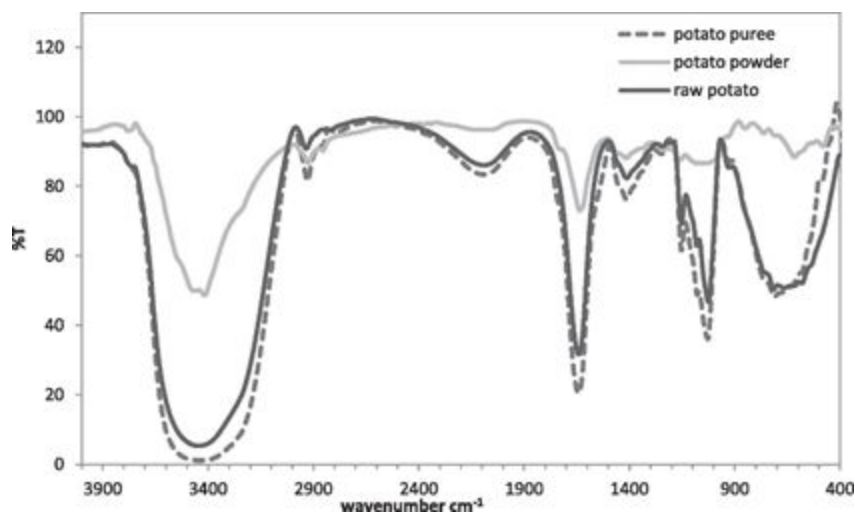


Figure 4.3.1. IR spectra for potato puree, potato powder, and raw potato [30]

The characteristic peaks for potato starch are at 3500 cm^{-1} , 2900 cm^{-1} , 2100 cm^{-1} and 1650 cm^{-1} . Peaks at 1650 cm^{-1} were assigned to water molecules absorbed and the stretching vibration of the C=O bond. The peak at 2100 cm^{-1} was due to water content. The peak at 2900 cm^{-1} is due to CH_2 deformation, and the peak at 3500 cm^{-1} is due to OH bonds in the molecule.

The authors also looked at how additives can change the FTIR spectra. **Figure 4.3.2** shows the spectra for several additives including glycerol.

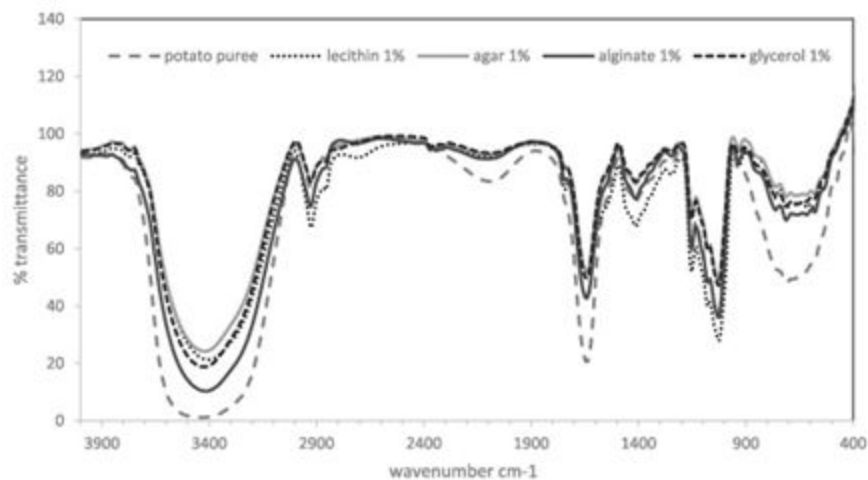


Figure 4.3.2. IR spectra for potato puree with additives [30].

With the glycerol, the potato starch shows the same peaks but with a lower intensity. The authors proposed that the additives disrupted the OH bond. This works to suppress the starch structure [26]. This confirms the idea of glycerol as a plasticizer. Plasticizers work by breaking down the intermolecular forces between polymer chains. They make plastics less rigid and brittle and more flexible. Potato starch plastic with no plasticizer is very brittle and shows poor mechanical properties. Adding glycerol helps to enhance our composites, allowing them to be used in more applications.

The FTIR spectra of the fibers used in the composites can be examined as well. Flax fibers are mostly composed of cellulose but also contain small quantities of hemicellulose, lignin, fat/wax, and water [31]. **Figure 4.3.3** shows the spectra for flax fiber. The study the spectrum comes from treated the flax fiber to increase adhesion between the fiber and matrix. The first spectra labeled untreated is what was used in our study.

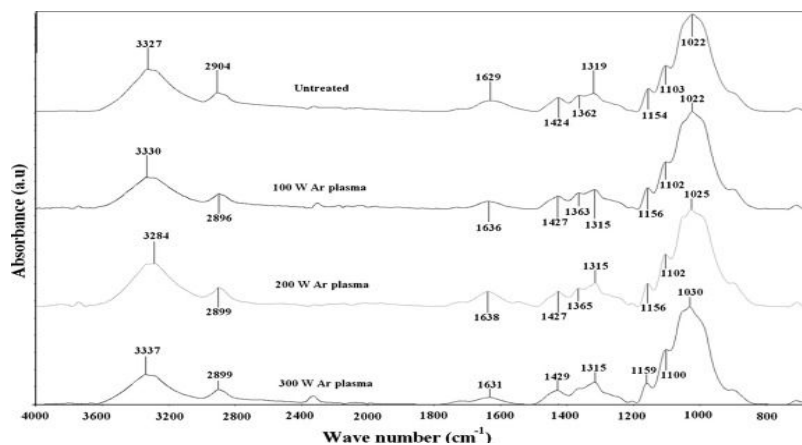


Figure 4.3.3 IR spectra for treated and untreated flax fibers [32]

There are several characteristic peaks for flax fiber. Bands in the range of 2750-3000 cm^{-1} are related to the CH stretching from CH and CH_2 in cellulose and hemicellulose. The peak around 1600-1640 cm^{-1} is likely associated with water in crystalline cellulose. The bands near 1362 cm^{-1} and 1319 cm^{-1} are due to CH_3 bending and CH_2 wagging in lignin. The tall peak at 1022 cm^{-1} is related to the C—O and O—H stretching vibration which belongs to the polysaccharide in cellulose [32].

4.4 Instron Testing

Once we determined the chemical composition of our composite samples, we next looked to investigate the mechanical properties of our sample. To do this, we looked to conduct tensile testing using an Instron machine. Specifically, a 2580 series, one hundred kilonewton static load cell would be used to carry out testing. This form of testing would allow us to gather information from a stress-strain curve such as the yield strength, Young's Modulus, and ultimate tensile strength.

As mentioned previously in *Section 2.7*, the ISO 524-7 testing standard is most suitable for our composite material. For chopped fiber-reinforced composites, this standard requires that the specimen be dogbone shaped. The mold that we initially created yields a small rectangular composite as seen in **Figure 4.4.1**. To obtain accurate measurements, we planned to laser cut or machine these rectangular specimens into the dogbone shape which would then be measured using calipers. Specifications such as gauge length, nominal width, and thickness are input into the testing software, and the instrument outputs a stress-strain curve upon completion of the testing procedure.



Figure 4.4.1 Rectangular mold, Instron testable composite samples - 0.5 g flax (left), 0.5 g jute (middle), 0.5 g cotton (right).

Unfortunately, we were unable to carry out the entire testing process due to the abrupt shutdown of campus facilities. However, we used our existing knowledge on the subject to make

reasonable predictions about what the results may have shown. The excel data obtained from the Instron test would yield a stress-strain curve similar to the one pictured in **Figure 4.4.2** [31]. This figure highlights the prototypical elements of a stress-strain curve that will be mentioned through the rest of this section. The linear region upon initially loading the material is known as the elastic region, while the slope of the elastic region is the Young's Modulus. The point at which material no longer displays elastic deformation is known as yield stress. The maximum stress that the material can experience before fracture is known as the ultimate tensile strength.

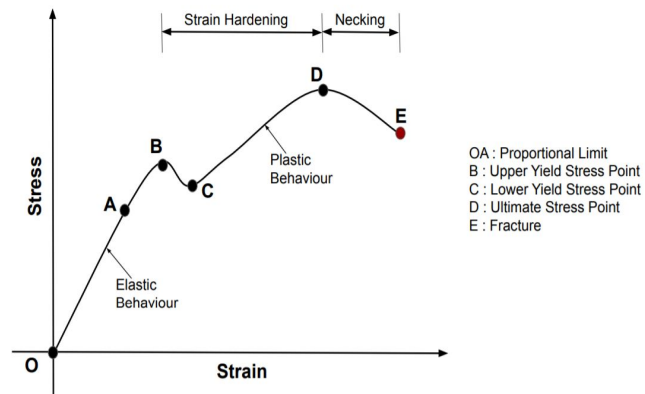
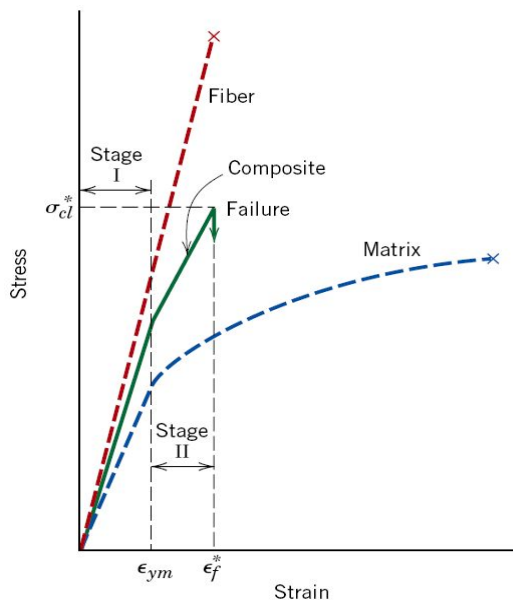


Figure 4.4.2. Prototypical stress-strain curve [33].

In fiber-reinforced composite materials, properties such as ultimate tensile strength, Young's Modulus, and yield strength depend largely on the characteristics of their individual fiber and resin components and how they interact. As seen in **Figure 4.4.3** [34], the stress-strain curve of the composite is roughly within the range of the fiber and matrix curves. The high Young's Modulus of the fiber component makes up for the lower Young's Modulus of the matrix component. The ultimate tensile strength is typically closer to that of the matrix curve, while the failure point depends heavily on the failure of the fiber component.



With this knowledge, we can predict that:

The Young's Modulus of our composite samples can be roughly characterized as the mean of the YM of its components. The ultimate tensile strength is roughly that of the matrix component. The yield strength approximately reflects that of the matrix component. The composite will not be as ductile as the matrix component; it will likely reflect the brittle nature of the fiber component, despite similar fracture loading as matrix.

Figure 4.4.3. Stress-Strain Curve - Composites [34].

The next step in our analysis was to research the properties of the fiber and matrix components we selected in our research. This fiber and resin matrix research can be summarized in **Tables 4.4.1** [35] and **4.2.2** [36, 37] below.

Fiber Component	Young's Modulus	Ultimate Tensile Strength
Flax	60-80 GPa	800-1500 MPa
Jute	10-30 GPa	400-800 MPa
Cotton	6 GPa	220 MPa

Table 4.4.1. Mechanical characterization of fibers [29].

Resin Component	Young's Modulus	Ultimate Tensile Strength
Potato Plastic	310 kPa	384 kPa
PLA	2004 MPa	41.8 MPa

Table 4.4.2. Mechanical characterization of resins [30, 31].

Although the resulting material properties of a material are largely dependent on the density of fibers within the resin matrix, we can use our additional research to predict the properties of a variety of fiber-matrix combinations (**Table 4.4.3**) This can only be done if we assume these hypothetical samples are isotropic and have 50% weightt fiber concentration.

	Young's Modulus	Ultimate Tensile Strength
Flax-Potato Starch	3.5 GPa	400 kPa
Jute-Potato Starch	1.0 GPa	400 kPa
Cotton-Potato Starch	3.0 GPa	400 kPa
Flax-PLA	35.1 GPa	42 MPa
Jute-PLA	10.1 GPa	42 MPa
Cotton-PLA	3.1 GPa	42 MPa

Table 4.4.3. Mechanical property prediction for composite samples.

4.5 Density

Our team speculated that samples with the lowest density would break down most rapidly. This is due to the fact that these low-density samples had ample space internal to the sample for microorganisms to inhabit. The first step in determining whether or not the density of the sample was related to the rate of degradation was to characterize our samples in terms of density. Unsurprisingly, the high flax composite had the lowest density at 0.787 g/mL. The low-density observed is likely attributed to the low density of the fibers, the high volume of low-density fibers, as well as the voids present in the composite sample which was cured under atmospheric pressure. The composite which was cured entirely in a low-pressure environment had the highest density at 3.5 g/mL. This is then followed by the plain potato polymer, the thin flexible composite, and the sample which was partially cured in a vacuum environment (**Figure 4.5.1**).

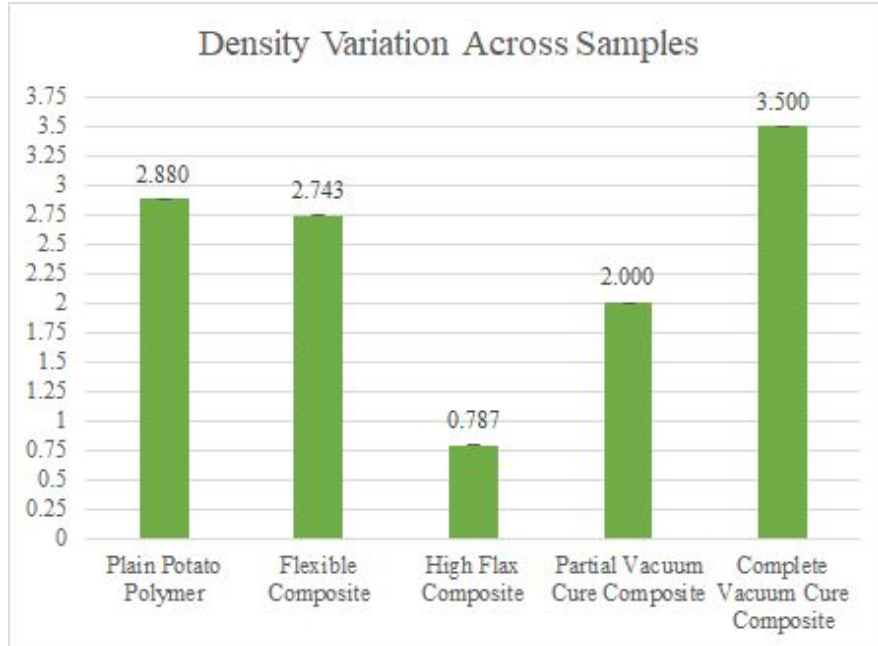


Figure 4.5.1: Density variation across samples.

4.6 Degradation

We observed the degradation of a variety of samples in a range of different environments over two months. By changing strategic variables, we were able to derive trends about how our different samples compared to one another, as well as how well they broke down in different environments.

The first environment we studied was that of a compost environment. This environment is characterized by moist soil and organic matter in the presence of microorganisms and worms. In this environment, we studied the rate of degradation of the plain potato polymer, the thin flexible flax composite, the high flax composite, the flax composite partially cured in a vacuum environment, and the flax composite entirely cured in a vacuum environment. We also included a sample of Vinyl Ester, which is utilized commercially, and is a common conventional matrix for composites made at home. The inclusion of Vinyl Ester allowed our team to draw comparisons between our potato-starch based composites, and commercially available matrices which can potentially be replaced with starch-based resins.

The sample with the greatest rate of mass loss in this environment was the high flax composite, which was also our sample with the lowest density. This is then followed by the thin flexible composite, the plain potato polymer, and then the composite which was partially cured in a vacuum environment. The sample with the slowest rate of degradation was the composite cured entirely in a low-pressure environment. These observed rates of degradation appear to closely correlate with the density of the sample which is being monitored. The only sample

which did not experience any mass loss associated with degradation was the Vinyl Ester, which we did not anticipate to degrade.

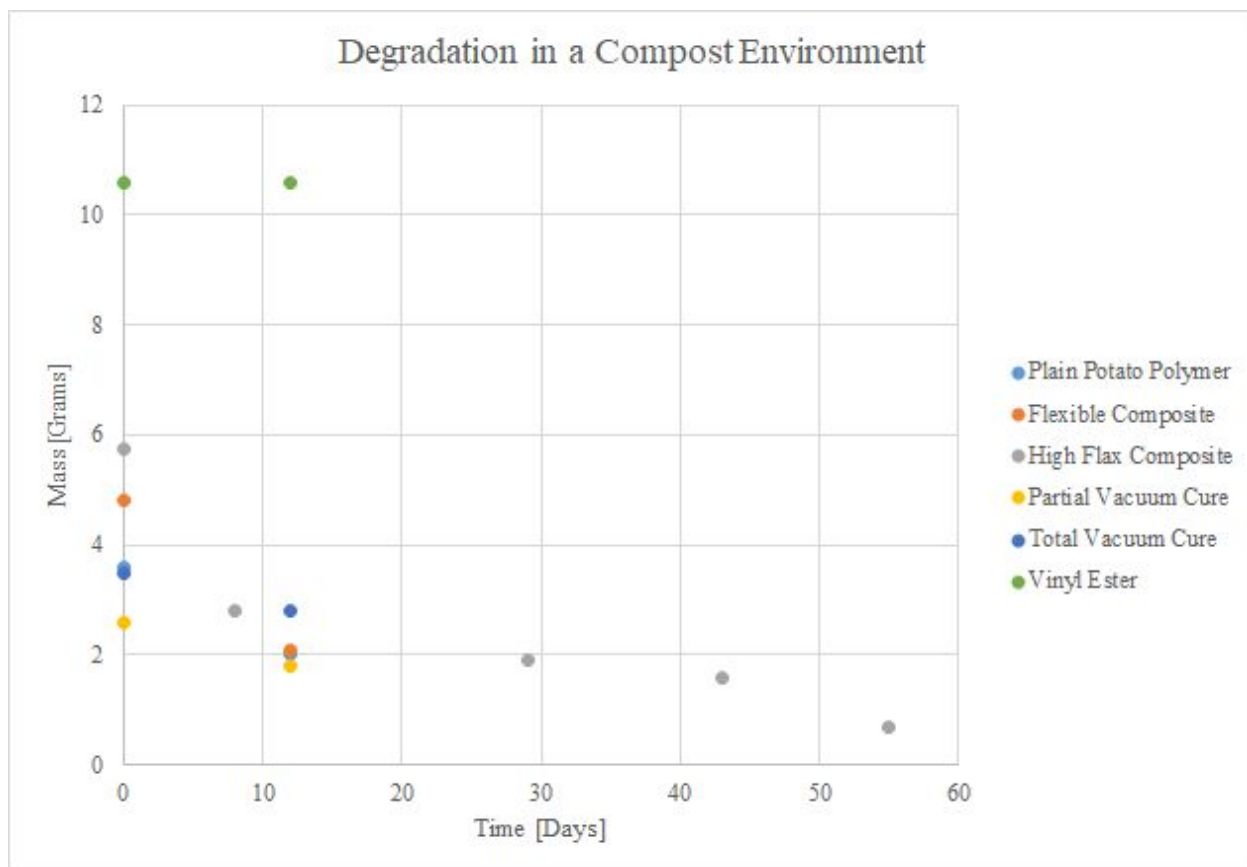


Figure 4.6.1: Degradation trends in a compost environment.

From these results, we were able to conclude that there is reasonable cause to believe that composite samples of this kind would degrade at rates that are related to their density when placed in compost environments.

The second component of the degradation study was to observe how environmental differences influenced the rate of degradation. For this study, we monitored the rate of mass loss of a high flax composite sample in ocean water, as well as a low oxygen environment. This is because compostable polymers have a high intrinsic ability to degrade, but this does not always translate to a high observed rate of degradation in non-ideal environments. We also took measurements on a sample which was sitting out in the open air to prove long term stability of the material, and to use as a baseline to compare the rate of degradation in other environments.

All of these high flax composite samples broke down at a faster rate than their counterparts in compost. Furthermore, we observed our ocean water sample become swollen and opaque, which is indicative of polymers breaking down into smaller chains. This suggests that our sample is truly compostable, even in saline environments. Additionally, we observed an

admirable rate of mass loss even in a low oxygen environment, which suggests that these composites will have some capacity to degrade even in landfill conditions (**Figure 4.6.2**)

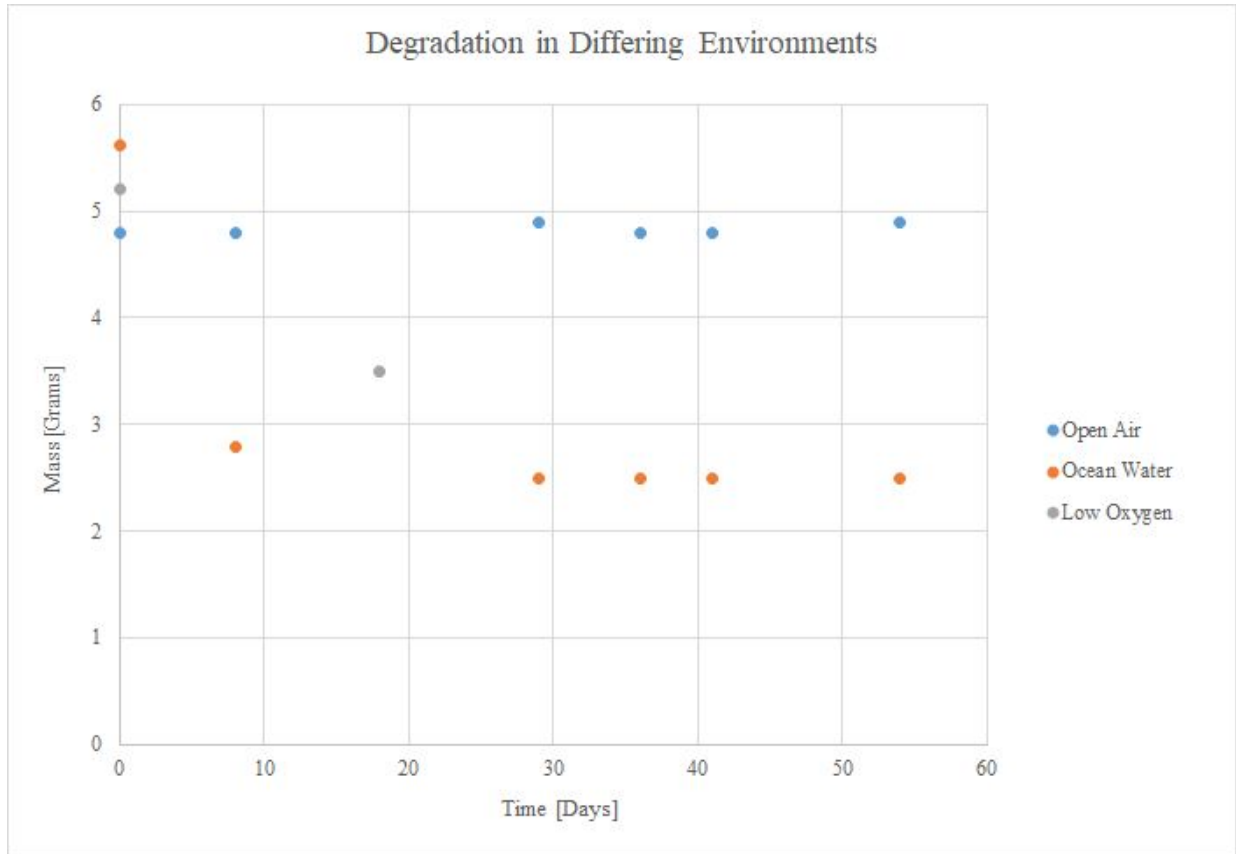


Figure 4.6.2: Degradation trends of a high flax composite in differing environments.

The rate of mass loss recorded was much higher in the first week after sample introduction into the environment, and drastically decreased as time went on (**Table 4.6.1**). This trend was observed with all samples in all environments. However, this was expected. According to our background research, we anticipated that the rate of decay for samples in an ideal environment would follow an Arrhenius equation, where the rate of mass loss would decline exponentially with respect to time. Our results roughly reflected this exponential decay trend, however, there were some fluctuations in conditions such as temperature and moisture content which prevented a true ideal degradation environment. For this reason, our results vary somewhat from mathematical predictions. This makes it difficult to predict when a sample will be completely degraded without running a longer degradation study.

Sample Environment	1 Week Mass Loss	4 Week Mass Loss
Ocean Water	50.18%	55.45%

Table 4.6.1: A representative sampling of mass loss rate declining over time. Here, mass loss numbers are referring to the mass lost, relative to the original mass of the sample, over a period of time.

4.7 Manufacturability

The method in which our samples were manufactured is known as casting. Casting is advantageous in that the production of samples is very quick, especially if the mold contains several operational cavities. Tooling costs involved in casting are low once the mold is created, but creation of a mold can be a costly process. Casting processes typically allow for high tolerances, however in the case of curing plastics in the mold cavity, the material can shrink below the initial design specifications. In the case of our products, samples shrunk to roughly one half of their initial height upon curing. Achieving highly precise design parameters involves greater raw material input and more complex machinery. Finally, the surface finish of the product is not its most desirable quality, but this worked to our benefit during testing since scanning electron microscopy requires rough surface textures.

4.8 Material Comparisons

To find a suitable application for our material, we compared the results from our material characterization to existing materials. This procedure was conducted in CES EduPack 2019 software using the Level 3 materials database. Two characteristics of particular interest to us were density and ultimate tensile strength. Using samples from our density analysis as well as estimations from our Instron analysis, we compared product density to ultimate tensile strength with the bubble chart seen in **Figure 4.8.1**. The CES Edupack software only provided data in units of lb/in³ (density) and kpsi (UTS), thus we converted our values in the excel table provided in **Appendix B**.

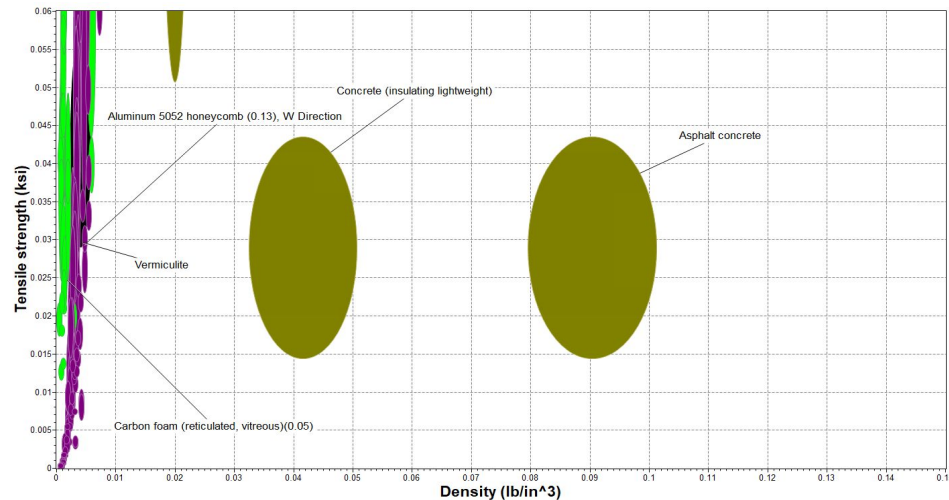


Figure 4.8.1. Material comparison using CES EduPack.

Information from this chart helps to understand the behavior of our material relative to other materials. Although our samples are unique, we found that materials that most closely resemble the strength-to-density ratios of our samples included carbon foam, vermiculite, aluminum 5052 honeycomb, concrete, and bricks. Of these materials, SEM imagery of bricks and concrete most closely resembles our SEM results, but our samples typically display lower void fractions overall. Closer synchronicity between our SEM imagery and the microscopic structures of commercial materials of similar density would serve as validation of our density estimations, but this conclusion cannot be drawn from our data.

4.9 Influence of Fiber Density

Differing fiber densities provided variation in material characteristics. We found that when the fiber density was low, the resulting composite had greater flexibility and malleability than higher fiber density composites. However, these low fiber density composites were also observed to be stronger than their plain potato polymer counterparts. When the fiber density was high, the composite was stiffer and sturdier. Fiber density alone did not determine the characteristics of the final composite - the curing method also had a profound impact on the resulting material. However, we observed distinct trends that relate the fiber density of the final composite to the characteristics of that composite. Some of our observations are detailed in **Table 4.9.1** “Characteristics of Different Composites.”

Sample	Fiber Density by Mass (Cured)	Characteristics
Plain Potato Polymer	0%	Flexible, Easy to Pierce or Rip
Thin Flexible Composite	2%	Strong, Flexible
High Flax Composite		Light, Malleable but Stiff
Partial Vacuum Cure Composite	20%	Moderately Stiff, Strong
Complete Vacuum Cure Composite	14.3%	Stiff, Rigid, Sturdy, Moderately Brittle

Table 4.9.1: Characteristics of different composites.

Like all composites, we found that there was a limit to which fibers no longer improved the structure and integrity of the material, but rather, hindered it. When the concentration of fibers within the composite was too high, it yielded a poor result. Among some of the defects associated with too high a fiber concentration include large voids in the matrix, poorly saturated fibers, and overall weakness of the sample. For this reason, it is very important to strategically select fiber densities based upon the intended usage of the product. Additionally, the upper threshold of these starch-based composites appears to be at approximately 30-33% by weight (**Figure 4.9.1**). This value varied based upon the fiber which was reinforcing the composite. This is similar to that of other Carbon Fiber Reinforced Composites, which typically have fiber saturation limits of 50-55% by volume.



Figure 4.9.1: Composite samples with fiber densities exceeding the saturation limit of the matrix.

Unfortunately, we were unable to calculate the exact fiber density of the High Flax Composite, because it was a cross-section of a larger sample. Additionally, the mass of fibers present in the sample was unknown. We were also unable to determine the void fraction within the sample, which would have allowed us to calculate the fiber density of the sample. Therefore, we were unable to draw conclusions about the influence of fiber density for that particular sample.

5.0 Other Findings

In this section, we describe results that we encountered over the course of the year that were critical to shaping this project's outcome but did not contribute directly to the main findings of the study. Our project changed trajectory several times throughout the year, and the procedures and findings listed below serve as examples of this. If time had permitted, each of these findings could have been explored further, as there was plenty left to discover from each.

5.1 PLA Composite Samples

PLA, as discussed in *Section 2.10*, would make a suitable material for the resin component of an organic fiber-reinforced composite. PLA was explored as an option during this project. First, PLA was obtained in the form of small, spherical pellets roughly 3mm in diameter. The first effort to form the plastic into a form that could be impregnated with fibers was baking the pellets in a large, rectangular mold. The melting point of PLA is approximately 220 C at most, so the pellets were placed into an open-face mold in an oven at 220 C for 1-2 hours. After several samples produced this way, it was determined that this method could not create a sample with low enough viscosity to be poured or otherwise combined with fibers after the baking process. The samples from this process, an example of which can be seen in **Figure 5.1.1**, also had many bubbles, likely due to the nature of the mold. It was difficult to remove the samples from the mold without freezing them first, as the melted PLA adhered to the mold after melting.

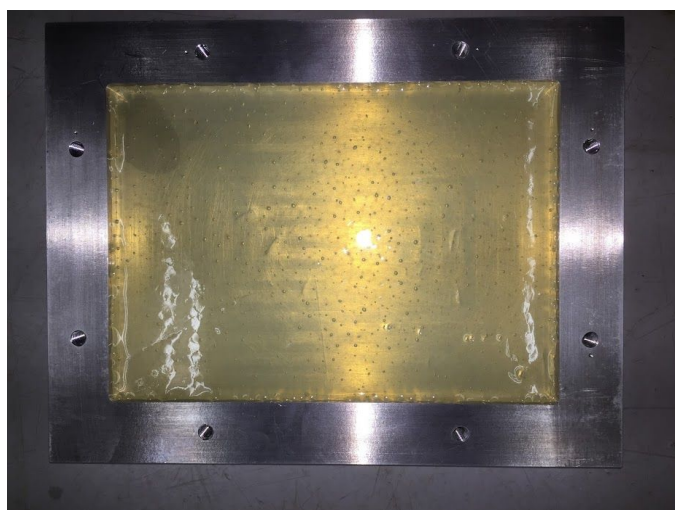


Figure 5.1.1. A sample of PLA pellets melted in an oven.

The next effort was to reduce the viscosity of the PLA. To do this, solvents with appropriate solubility parameters for PLA, and which were safe to use in the lab, were added to a

small sample of pellets and placed on a hot plate. The solvents chosen were tetrahydrofuran and ethyl acetate. The intent was to lower the melting temperature of the PLA and ultimately increase the viscosity of the melted PLA by using the solvents to help break the secondary bonds of the molecular chains in the plastic. However, samples produced by this method as exemplified in **Figure 5.1.2**, did not completely melt. Thus, this process was discontinued.



Figure 5.1.2. A sample of PLA pellets melted in the presence of solvents.

To address the issues of bubbles and inconsistent melting, pellets were pulverized using a Kitchenaid immersion blender. Once added to a mold, we anticipated that the pulverized PLA had much less void space than the pellets, since the larger spheres of pellets did not fit together as well as the much smaller flakes of pulverized PLA. One major constraint to this method was the production time of pulverized PLA; to fill the small rectangular mold entirely with PLA granules, we were required to blend pellets for over thirty minutes. This alone played a large role in the discontinuation of this method.

The blending process took place in a metal beaker since blending in a plastic or glass container would ultimately shatter it. Since the pulverization rate was slow, PLA granules were mixed among larger intact pellets, making it difficult for us to filter out our product. To troubleshoot this issue, we conducted a vacuum pump filtration method using water and coffee filters to separate pellets from for granule products.

As previously mentioned, we next looked to add our pulverized PLA to the small mold and melt it accordingly. Rather than oven melting, we looked for a more direct melting approach, one that directly heated the top and bottom face of the mold for maximizing conductive heat transfer. The solution to our problem was a hot press machine, located at the PracticePoint labs in Gateway Park, which melted our samples both uniformly and quickly. It was difficult for us to

intersperse fibers randomly within the pulverized PLA, so we chose to layer fibers on a two-dimensional, horizontal plane roughly halfway through the sample's height. The resulting layered PLA samples are pictured below in **Figure 5.1.3**.



Figure 5.1.3. Flax-reinforced (0.5 g), hot pressed PLA sample.

As seen in the bottom view image, the sample was highly imperfect due to adhesion between the PLA and aluminum during the curing process. As a result, we did not carry on with further testing of the PLA sample, but with skillful machining, it is very possible that this sample could also be mechanically tested with an Instron machine.

5.2 Polymerization of Polylactic Acid

Another method that was attempted early on in the project was to polymerize PLA from lactic acid instead of melting PLA pellets and adding fibers to them. One of the main issues with melting PLA in an oven was that it was too viscous to put fibers in. We anticipated that by conducting the polymerization process, the PLA would be less viscous, and fibers would be able to be mixed in. In order to polymerize the PLA, 85 wt% lactic acid was used with stannous octoate as a catalyst. First, lactic acid was dehydrated in a rotary evaporator, or “rotovap” for short, at 160 C for 2 hours. The temperature was then increased to 220 C with a reduced pressure of 200 mmHg for about 4 hours. The condenser was maintained at 90 C to ensure it did not solidify. Lactide was obtained by distillation and the solid product in the condenser was washed and dried overnight. The lactide was then mixed with the catalyst at 140 C for two hours to make the PLA [32].

Several issues prevented us from polymerizing the PLA. One of the issues was that the heater for the rotovap was unable to reach the temperatures required for the polymerization. This was solved by using a bowl of silicone oil on a hot plate which was monitored using a temperature gun. Our final rotovap configuration can be seen in **Figure 5.2.1**. The next issue was that, even at the correct temperature, there was no evaporation or condensation of the water from the lactic acid. This was due to dry rot on the tube between the vacuum pump and the rotovap. An insufficient vacuum may have prevented the evaporated water from condensing out. A usable

sample of PLA was not able to be obtained. One of the first runs resulted in some lactide which was mixed with the catalyst. A small amount of PLA was made, but it was not enough to create a composite sample. In the future, the dry rotted tubing for the rotovap should be replaced, or a distillation column could be used for the dehydration and lactide collection.



Figure 5.2.1. Rotary evaporator hood configuration.

6.0 Recommendations

Based on our findings, several recommendations were developed to provide future researchers with a variety of avenues to explore on this topic. These recommendations range from direct continuations of research methods cut short by the COVID-19 pandemic, as well as points for improving upon our research.

6.1 Recommendation One

We recommend characterizing the mechanical properties of potato starch-based fiber-reinforced composites using the Instron testing method.

In order to properly characterize the mechanical properties of these fiber-reinforced composites, Instron testing is necessary. The most direct method to obtain a specimen for this test would be to create a sample in a rectangular mold, such as the example in *Section 3.2*, and to then machine or laser-cut the properly dimensioned test subject from the rectangular specimen. An Instron test, as detailed in *Sections 2.7* and *3.7*, will provide data regarding the material's yield strength, ultimate tensile strength, modulus of elasticity, and more. Values obtained from this test will serve as a platform on which other materials can be compared. If a material has a similar profile of mechanical properties and is sourced from something such as crude oil, these composites may be offered as a less environmentally detrimental alternative. Perhaps the properties of the material may not match any existing materials well and could be used for new applications instead. Whatever the case, Instron testing should be conducted in order to make informed predictions about the behavior of the material.

6.2 Recommendation Two

We recommend identifying the composition of potato starch-based fiber-reinforced composites via FTIR analysis.

Another test that would yield valuable insight is the FTIR test, as described in *Section 3.6*. FTIR testing yields results pertaining to the molecular composition and structure of organic materials. Since these particular fiber-reinforced composites are made from organic materials, an FTIR test can be used to analyze their entire makeup. While the results of this test may not be as directly related to potential applications as Instron testing, it is valuable in its description of the results of the manufacturing process. Samples may change based on which type of fiber is used, how much of the fiber was used, and what environment the plastic cured in. Furthermore, the processes described in the methods chapter can be altered to produce new results, and the FTIR test is one of the best indicators of those results.

6.3 Recommendation Three

We recommend applying several composite samples produced in this study to real-world applications.

Although further investigation of the mechanical properties of the samples would allow for more informed recommendations for applications, the information collected via degradation analyses, SEM imagery, and laboratory observations provide enough to form basic assumptions about potential applications. For example, one potential application for our 0.25 g flax-reinforced potato starch-based thin and flexible composite is a plastic grocery bag. The high void fraction and compostability over a two month period make it a great replacement for single use plastics. Additionally, the 0.25 g flax-reinforced PLA-based hard, high strength plastic could be applied to a children's toy. Both of these products contribute to the waste crisis, and using our composite as a replacement material, significant steps can be made toward improving our environmental situation.

6.4 Recommendation Four

We recommend curing fiber-reinforced composites under vacuum conditions to minimize voids within the sample.

One major finding from our SEM analysis was the existence of voids within our composite samples. Initially, we proposed the hypothesis that increased voids result in faster degradation, and the results of our testing suggest that this is true when compared to the composite degradation within a low-pressure environment. Thus, samples with more uniform microscopic structures must be created, and consequently applied to even more new applications.

Due to halted data collection in D-term, we were unable to synthesize more than one vacuum-cured sample. However, the existence of our one vacuum-cured flax-reinforced potato-based composite shows that this method of sample creation is possible. To achieve this, one must create a small vacuum vessel equipped with a vacuum gauge and hose. Machine shop and lab manager, Tom Partington assisted in the creation of this vessel. The vessel we used consisted of eight hex bolts, washers, and nuts to seal the lid while curing. Once the composite is prepared and enclosed within a low vacuum environment (mmHg), we recommend leaving the sample to cure for approximately three to five days for optimal results.

6.5 Recommendation Five

We recommend creating future samples using unidirectional or woven fabric fiber orientations.

Our study was mainly focused on determining the most effective fiber quantity within our composite sample, and in doing so, we exclusively used random fiber orientations to minimize process variables. As we mentioned in *Section 2.6*, altering the fiber orientation could also have

an impact on the properties of the product, so we recommend studying this particular variable in greater depth. To do this, we recommend keeping the fiber density uniform across all samples, and specifically, samples with 0.5 g of fiber cured best for us. Using samples of 0.5 g fiber density, we propose aligning unidirectional and woven fibers within the resin matrix for comparison to our random, chopped fiber products. Although these composites are more difficult to produce, our research suggests that properties such as tensile strength, stiffness, and impact absorptivity will be enhanced, and the composites can be used in a broader spectrum of applications as a result.

6.6 Recommendation Six

We recommend using new resin materials, specifically developing corn starch-based fiber-reinforced composites.

Finally, we recommend altering the resin material within the composite, and instead of creating potato starch-based composites, cornstarch-based resins could be a reasonable alternative. Corn starch is a readily available, inexpensive resource, and its implementation within fiber-reinforced composites could greatly impact matrix-fiber adhesion seen at a microscopic level. This can be tested using SEM analysis much like we conducted in our study. A degradation study over several months could also be conducted using corn-starch based composites in open-air natural environments, high salinity bodies of water, and anaerobic environments. Results from this degradation study can be compared to those collected in this study to determine the relative compostability of the composite.

7.0 Conclusion

Modern society's reliance on synthetic, petroleum-based plastics is one of the main contributing factors to the waste crisis. Biologically sourced, biodegradable plastics are an emerging industry that provides one solution to the waste crisis. In this project, a novel composite material made from biodegradable resin and natural fibers was developed and studied.

Composite samples were made with plastic made from potato starch and either jute, flax, or cotton. SEM pictures of the samples were taken. Flax and cotton showed the best fiber-matrix adhesion while flax and jute had pores which could improve degradability and impede crack propagation. While Instron testing was not able to be completed, predictions of Young's modulus and ultimate tensile strength were calculated. The three potato plastic composites had the same ultimate tensile strength of 400 kPa. The flax sample had the highest Young's modulus, 3.5 GPa, while the jute sample had the lowest, 1.0 GPa. The density of the samples was also determined. The high flax composite had the lowest density while the vacuum-cured composite had the highest. It was speculated that lower density composites would degrade quicker due to a higher void fraction. This was supported by the degradation study performed. The flax composite degraded the quickest while the vacuum-cured composite degraded the slowest in compost. Degradation was also studied in non-ideal environments such as ocean water and a low-oxygen environment to simulate a landfill. The samples showed the ability to degrade in saline and landfill conditions which supports that the composite is truly compostable.

There are several recommendations we developed for the future study of this topic. We recommend further study of the mechanical and chemical properties of the composite using the Instron testing method and ATR-FTIR method, respectively. These analyses could then be compared to the predictions made in this paper. The results of these tests could also help to identify and develop uses for potato plastic composites including packaging, shopping bags, and parts for toys. Finally, we recommend studying different methods of production. Our project was limited to potato starch plastic, random fiber orientation, and oven-cured composites. There are many more options that could be studied including cornstarch-based plastic, woven fibers, and vacuum curing of the plastic.

8.0 References

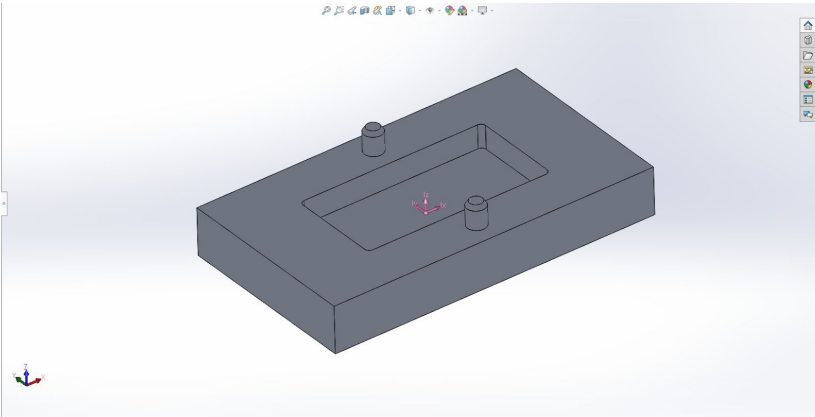
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Appendix A: Mold Design



Base Design

Mass Properties

base.SLDPRT

Options...

Override Mass Properties... Recalculate

☒ Include hidden bodies/components

☐ Create Center of Mass feature

☐ Show weld bead mass

Report coordinate values relative to: -- default --

Mass properties of base

Configuration: Default

Coordinate system: -- default --

Density = 0.10 pounds per cubic inch

Mass = 2.35 pounds

Volume = 24.12 cubic inches

Surface area = 85.04 square inches

Center of mass: (inches)

X = 3.50

Y = 2.00

Z = 0.46

Principal axes of inertia and principal moments of inertia: (pounds * square inch

Taken at the center of mass.

lx = (1.00, 0.00, 0.00) Px = 3.74

ly = (0.00, 1.00, 0.00) Py = 10.83

lz = (0.00, 0.00, 1.00) Pz = 14.17

Moments of inertia: (pounds * square inches)

Taken at the center of mass and aligned with the output coordinate system.

Lxx = 3.74 Lxy = 0.00 Lxz = 0.00

Lyx = 0.00 Lyy = 10.83 Lyz = 0.00

Lzx = 0.00 Lzy = 0.00 Lzz = 14.17

Moments of inertia: (pounds * square inches)

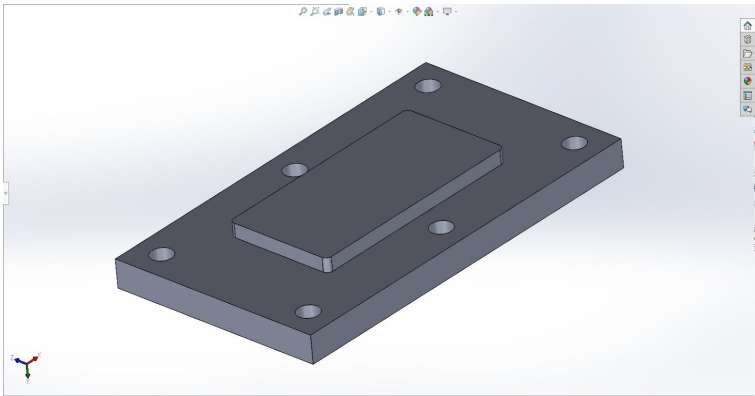
Taken at the output coordinate system.

lxx = 13.65 lxy = 16.47 lxz = 3.81

lyx = 16.47 lyy = 40.16 lyz = 2.18

lzx = 3.81 lzy = 2.18 lzz = 52.41

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Cover Design

Mass Properties

cover.SLDPRT

Options...

Override Mass Properties... Recalculate

☒ Include hidden bodies/components

☐ Create Center of Mass feature

☐ Show weld bead mass

Report coordinate values relative to: -- default --

Mass properties of cover

Configuration: Default

Coordinate system: -- default --

Density = 0.10 pounds per cubic inch

Mass = 1.52 pounds

Volume = 15.62 cubic inches

Surface area = 72.22 square inches

Center of mass: (inches)

X = 3.50

Y = 0.20

Z = -2.00

Principal axes of inertia and principal moments of inertia: (pounds * square inch

Taken at the center of mass.

lx = (1.00, 0.00, 0.00) Px = 1.86

ly = (0.00, 0.00, -1.00) Py = 5.67

lz = (0.00, 1.00, 0.00) Pz = 7.42

Moments of inertia: (pounds * square inches)

Taken at the center of mass and aligned with the output coordinate system.

Lxx = 1.86 Lxy = 0.00 Lxz = 0.00

Lyx = 0.00 Lyy = 7.42 Lyz = 0.00

Lzx = 0.00 Lzy = 0.00 Lzz = 5.67

Moments of inertia: (pounds * square inches)

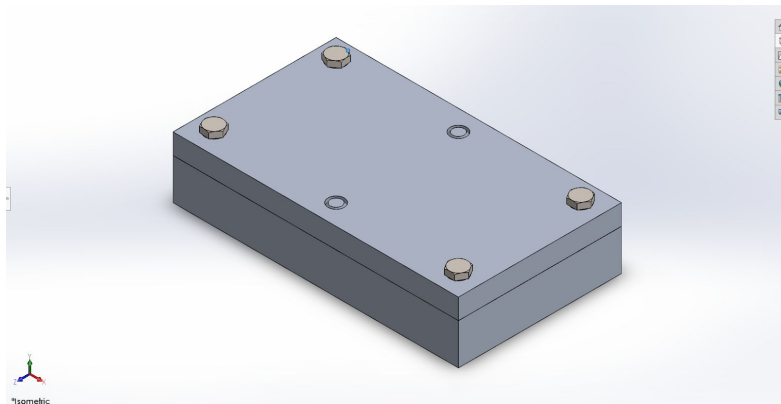
Taken at the output coordinate system.

lxx = 8.01 lxy = 1.08 lxz = -10.67

lyx = 1.08 lyy = 32.18 lyz = -0.62

lzx = -10.67 lzy = -0.62 lzz = 24.39

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Assembly Design

Mass Properties

Mold Assembly.SLDASM

Options...

Override Mass Properties... Recalculate

☒ Include hidden bodies/components

☐ Create Center of Mass feature

☐ Show weld bead mass

Report coordinate values relative to: -- default --

Mass properties of Mold Assembly
Configuration: Default
Coordinate system: -- default --

Mass = 3.88 pounds

Volume = 39.95 cubic inches

Surface area = 161.03 square inches

Center of mass: (inches)
X = 3.26
Y = 3.22
Z = 5.08

Principal axes of inertia and principal moments of inertia: (pounds * square inches)
Taken at the center of mass.
I_x = (1.00, 0.00, 0.00) P_x = 6.12
I_y = (0.00, 0.00, -1.00) P_y = 17.08
I_z = (0.00, 1.00, 0.00) P_z = 21.67

Moments of inertia: (pounds * square inches)
Taken at the center of mass and aligned with the output coordinate system.
L_{xx} = 6.12 L_{xy} = 0.00 L_{xz} = 0.00
L_{yx} = 0.00 L_{yy} = 21.67 L_{yz} = 0.00
L_{zx} = 0.00 L_{zy} = 0.00 L_{zz} = 17.08

Moments of inertia: (pounds * square inches)
Taken at the output coordinate system.
I_{xx} = 146.40 I_{xy} = 40.72 I_{xz} = 64.28
I_{yx} = 40.72 I_{yy} = 163.05 I_{yz} = 63.42
I_{zx} = 64.28 I_{zy} = 63.42 I_{zz} = 98.53

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Appendix B: Tensile Strength Conversion

Density and Ultimate Tensile Strength Unit Conversions Table:

	Density		UTS	
	g/mL	lb/in ³	kPa	kpsi
Plain Potato Polymer	2.58	0.104	400	0.058
Flexible Polymer	2.74	0.098	400	0.058
High Flax Ploymer	0.757	0.028	400	0.058
Vacuum Sample 1	2	0.072	400	0.058
Vacuum Sample 2	3.5	0.126	400	0.058